

Laser flash photolysis of sodium persulfate in aqueous solution with additions of dimethylformamide

K.L. Ivanov^a, E.M. Glebov^b, V.F. Plyusnin^{b,*}, Yu.V. Ivanov^b, V.P. Grivin^b, N.M. Bazhin^b

^a Novosibirsk State University, 630090, Novosibirsk, Russia

^b Institute of Chemical Kinetics and Combustion SB RAS, 630090, Novosibirsk, Russia

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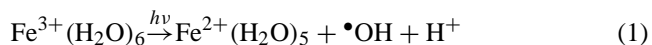
Abstract

The method of laser flash photolysis with excitation at 308 nm was used to study the photochemistry of persulfate-ion in aqueous solutions with additions of *N,N*-dimethylformamide (DMF). The photolysis of $S_2O_8^{2-}$ in water gives rise to $SO_4^{\bullet-}$ radical anions with a 0.55 quantum yield. The rate constant of $SO_4^{\bullet-}$ recombination was measured ($2k = (2.7 \pm 0.2) \times 10^9 M^{-1} s^{-1}$ at ionic strength $\mu = 0.9 M$). In the presence of DMF the additional intermediate absorption band appears with a maximum at 380 nm, belonging to radical $\bullet CH_2(CH_3)NCHO$. Its extinction coefficient was determined ($\epsilon^{380} = 1700 \pm 200 M^{-1} cm^{-1}$). The rate constants of reactions $SO_4^{\bullet-} + DMF ((4.3 \pm 0.2) \times 10^8 M^{-1} s^{-1})$ and $\bullet CH_2(CH_3)NCHO + O_2 ((1.3 \pm 0.1) \times 10^9 M^{-1} s^{-1})$ were measured. Thus, DMF is a good acceptor of active radicals (e.g., $\bullet OH$ radical) which have absorption in the far UV region and which are impossible to detect directly in pulse photochemical experiments. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Persulfate; $SO_4^{\bullet-}$; Dimethylformamide; Laser flash photolysis

1. Introduction

Water in oceans, rivers, and rain drops contains a great number of organic and inorganic impurities which participate in various photochemical and thermal reactions. Of major importance in the reactions of natural water systems are the processes, initiated by a hydroxyl radical [1]. It is assumed that the most significant source of $\bullet OH$ radicals in natural water are the photochemical reactions of the complexes of trivalent iron, in particular, of $Fe^{3+}(OH^-)$ and Fe_{aq}^{3+} [2,3]. Hydroxyl radicals are considered to appear in the first co-ordination sphere of the excited $Fe(III)$ complexes due to electron transfer from either OH^- group or water molecule [4]:



However, conclusion about $\bullet OH$ radical formation is based on stationary experiments, mainly, on the analysis of the final photolysis products [3–8]. The direct pulse experiments, confirming the formation of this radical, are unavailable in the literature due to the fact that $\bullet OH$ radical is difficult to record by optical methods. The most long-wave

band of its absorption has a maximum at 225 nm with extinction coefficient $\epsilon^{225} \approx 600 M^{-1} cm^{-1}$ [9] which, as a rule, is masked by the absorption of initial iron complexes. Therefore, it is necessary to use a trap, i.e. a compound which reacts with hydroxyl radical to form an intermediate species whose optical absorption spectrum is convenient for recording, to detect $\bullet OH$ radical in pulse photochemical experiments.

Amides seems to be of interest as such traps. It has been shown using pulse radiolysis [10] that amides rapidly (rate constants are about $10^9 M^{-1} s^{-1}$) react with $\bullet OH$ radical giving transient species which have optical absorption bands in the region of about 380 nm with extinction coefficients of about $2000 M^{-1} cm^{-1}$. These transient species were assigned in [10] as organic radicals. For *N,N*-dimethylformamide (DMF) *N*-methyl-*N*-methylene-formamide radical $\bullet CH_2(CH_3)NCHO$ is formed



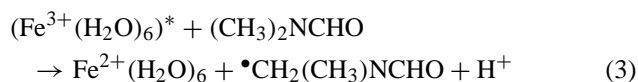
Such an assignment is supported by the experiments on low-temperature radiolysis of amides with the recording of both optical and ESR spectra [11].

Recently [12] an attempt has been made to use DMF as the radical trap in studying the photochemistry of Fe_{aq}^{3+} . It has been shown that the flash photolysis of the

* Corresponding author.

E-mail address: plyusnin@ns.kinetics.nsc.ru (V.F. Plyusnin)

oxygen-free aqueous solutions of $\text{Fe}_{\text{aq}}^{3+}$ with added DMF gives rise to intermediate absorption with a maximum at 380 nm. The spectrum of this intermediate coincides with that of *N*-methyl-*N*-methylene-formamide radical $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ [10,11]. There are two possible processes, leading to the formation of this radical. First, this is the reaction of hydroxyl radical with DMF (Reaction 2). Another possibility is related to electron transfer from DMF molecule to the light-excited complex:



A similar process is typical of the photochemistry of copper(II) chloride complexes in DMF solutions [13,14].

In order to use DMF as a trap of primary radical particles in experiments on the photochemistry of co-ordination systems in aqueous solutions, it is necessary to have information about the rate constants of reactions involving the $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical. The rate constant of its recombination is the only one available in the literature [10,12]). The goal of this paper is to determine the rate constants of the reactions of radical formation and decay. To obtain high concentrations of these radicals we used persulfate-ion ($\text{S}_2\text{O}_8^{2-}$) solutions with added DMF. In these systems the radicals are produced with a higher quantum yield than upon the photolysis of Fe^{3+} complexes.

2. Experimental details

The $\text{S}_2\text{O}_8^{2-}$ solutions were prepared using the three-fold distilled water purified of organic impurities and sodium persulfate. Experiments were carried out in neutral and acid media; solution pH was varied by addition of perchloric acid. The solution ionic strength was supported by $\text{Na}_2\text{S}_2\text{O}_8$ and varied in the range of 0.3–0.9 M (up to 0.3 M $\text{Na}_2\text{S}_2\text{O}_8$). When necessary, the samples were bubbled through by nitrogen to remove oxygen. All experiments were performed at room temperature (20°C). The optical absorption spectra were recorded using a Specord UV–VIS (Carl Zeiss) spectrophotometer. Experiments on laser flash photolysis were carried using an XeCl laser excitation (308 nm) [15]. In all experiments we used quartz cuvettes with a 1 cm optical path. The product of quantum yield by extinction coefficient of intermediate absorption ($\varepsilon\varphi$) upon photolysis was measured by comparing the experimental signal with the standard signal of T–T absorption at 431 nm of the oxygen-free anthracene solution in benzene ($\varepsilon\varphi=22260$ [16]).

3. Results and discussion

3.1. Photolysis of aqueous solutions of $\text{Na}_2\text{S}_2\text{O}_8$

The optical absorption spectrum of $\text{S}_2\text{O}_8^{2-}$ ion in aqueous solutions has a band with a maximum at 215 nm and the

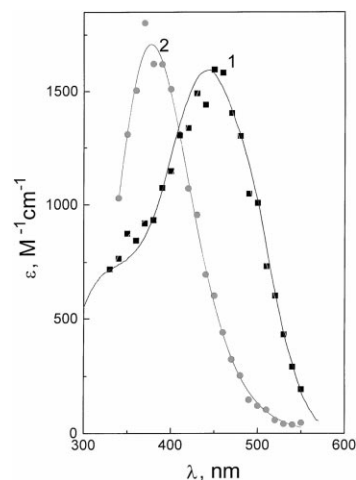


Fig. 1. Electron absorption spectra of $\text{SO}_4^{\bullet-}$ radical anion (1) and $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical (2). The spectrum of $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical is obtained for solutions with DMF concentration equal to 10^{-2} M. The solid line (1) is the $\text{SO}_4^{\bullet-}$ spectrum taken from Ref. [23].

extinction coefficient $\varepsilon^{215} = 220 \text{ M}^{-1} \text{ cm}^{-1}$ (with 308 nm, $\varepsilon^{308} = 1.2 \text{ M}^{-1} \text{ cm}^{-1}$). It is well known [17,18] that the $\text{S}_2\text{O}_8^{2-}$ photolysis gives $\text{SO}_4^{\bullet-}$ radical anions, displaying the optical absorption band with a maximum at 455 nm [17–19]:



The spectrum of intermediate absorption obtained by us upon laser flash photolysis of sodium persulfate in water (Fig. 1, curve 1) coincides with the published one and belongs to the $\text{SO}_4^{\bullet-}$ radical anion. The product of extinction coefficient of the absorption band of $\text{SO}_4^{\bullet-}$ radical anion by the quantum yield of its formation was determined to be $\varepsilon^{455}\varphi^{308} = 880 \pm 80 \text{ M}^{-1} \text{ cm}^{-1}$.

The extinction coefficient of the band at 455 nm was repeatedly measured [17–23] and the published values vary from 460 [18] to $1600 \text{ M}^{-1} \text{ cm}^{-1}$ [21–23]. The latest papers give, as a rule, the values close to $1600 \text{ M}^{-1} \text{ cm}^{-1}$. Therefore, a given value is considered the closest to the real extinction coefficient [24,25]. In [19,22] the authors discuss the possible reasons for the underestimation of extinction coefficient. We use this value to determine both the quantum yield of $\text{S}_2\text{O}_8^{2-}$ photodissociation ($\varphi^{308\text{nm}} = 0.55 \pm 0.05$) and the rate constant of $\text{SO}_4^{\bullet-}$ radical anion decay.

In [19] the method of laser flash photolysis with excitation at 248 nm was used to measure the value of $\varepsilon^{455}\varphi^{248} = 2770 \text{ M}^{-1} \text{ cm}^{-1}$. The quantum yield of radical formation is assumed to be 2. A specified value of extinction coefficient ($\varepsilon^{455} = 1600 \text{ M}^{-1} \text{ cm}^{-1}$) makes it possible to obtain the value of $\varphi^{248} = 1.73$ using the data of paper [19]. A smaller value of quantum yield upon photolysis by a more long-wave light ($\varphi^{308} = 0.55$) is likely to be related to both a smaller distance in a radical pair after thermalization and the greater probability of geminate recombination.

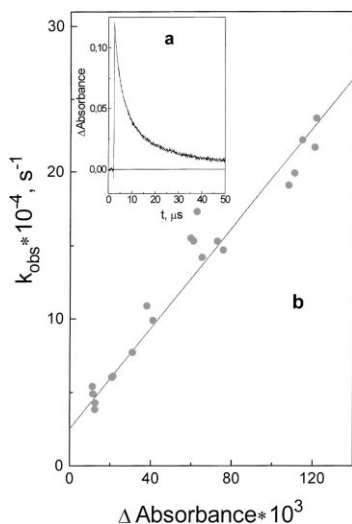
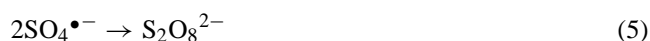


Fig. 2. Laser flash photolysis of Na₂S₂O₈ (0.3 M) in water. (a) An example of a kinetic curve at 455 nm; (b) the dependence of k_{obs} of SO₄^{•-} radical anion decay on the initial optical density at 455 nm.

Fig. 2a exemplifies the experimental kinetic curve of the intermediate optical absorption of radical SO₄^{•-} decay. The radical decay rate depends on the initial radical concentration which may be varied by changing the intensity of the exciting laser pulse. The dependence of the observed effective first-order rate constant determined by the initial region (10–20% of total amplitude) of kinetic curves ($k_{\text{obs}} = -(\partial D/\partial t)_{t=0}/D_{t=0}$) where D is the optical density of intermediate absorption on the initial radical concentration is linear (Fig. 2b). Thus, the kinetics of SO₄^{•-} radicals decay is well described by the second-order curve, and the basic process causing their decay in the system studied is recombination:



Using a $1600 \text{ M}^{-1} \text{ cm}^{-1}$ extinction coefficient of the SO₄^{•-} radical band, the constant $2k_5$, determined from the slope of the straight line in Fig. 2b, is $2k_5 = (2.7 \pm 0.2) \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$. The value of this rate constant was repeatedly measured by various methods [18–20,26–30]. Fig. 3 gives these data using the typical dependence of the reaction rate constant on ionic strength (μ) for charged particles [19]:

$$\log(k_5) \approx \log(k_5^0) + 1.02 z_1 z_2 \frac{\mu^{1/2}}{1 + \mu^{1/2}} \quad (6)$$

In Formula (6) z_1 and z_2 are the charges of reacting ions. So our value for the rate constant of SO₄^{•-} radical anion recombination agrees satisfactorily with the majority of literature data. It is necessary to stress that the recent measurements of Herrmann et al. [29] have given the value of $2k_5 = 3.2 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$ (at $\mu=0$) which is much lower than the other data available. May be, the authors of [29] have used the overestimated value of the extinction coefficient of SO₄^{•-} ($1090 \text{ M}^{-1} \text{ cm}^{-1}$ at 250 nm).

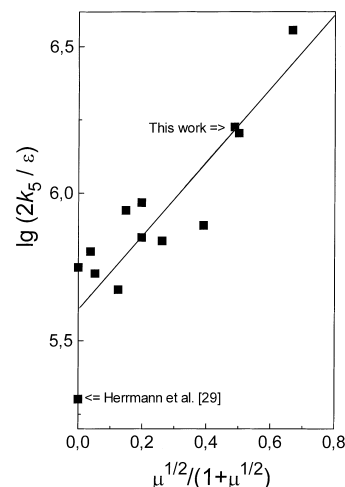
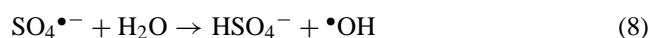
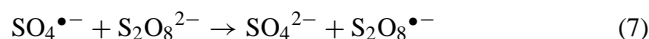


Fig. 3. The dependence of a $2k_5/\epsilon^{455}$ ratio on the ionic strength of solution for SO₄^{•-} radical anion recombination according to [16,18–20,25,26,34] and this work.

The cut-off on the ordinate (Fig. 2b) corresponds to the contribution of the pseudo-first order reaction to the decay of SO₄^{•-} radical anions which may correspond to the reactions with persulfate ion and water:



In the presence of reaction (7) the pseudo-first order rate constant is sure to increase linearly with increasing concentration of a persulfate ion. Measuring the dependence of the cut-off value on S₂O₈²⁻ concentration with regard to the ionic strength of the solvent according to Eq. (6) (Fig. 4), we have estimated the contribution of reaction (7): $k_7 = (5.3 \pm 2.0) \times 10^3 \text{ M}^{-1} \text{ s}^{-1}$ (note that the value obtained is

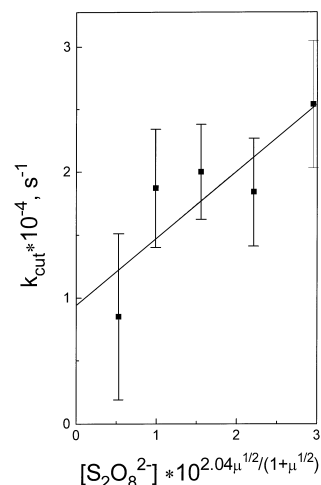


Fig. 4. Reaction of SO₄^{•-} radical anion with persulfate-ion and water. The dependence of k_{cut} (the cut-off on the ordinate in Fig. 2b) of SO₄^{•-} radical anion decay on S₂O₈²⁻ concentration. The dependence of the rate constant on ionic strength (Eq. (6)) is taken into account.

related to zero ionic strength). The cut-off on the ordinate in Fig. 4 is, probably, determined by reaction (8) and estimates $k_8 = (9.4 \pm 3.7) \times 10^3 \text{ s}^{-1}$. This gives for the bimolecular constant of reaction (8) the value of $170 \pm 70 \text{ M}^{-1} \text{ s}^{-1}$.

The value of k_7 obtained in this work is much lower in comparison with the previous measurements [23,24,29,31,32]. It is surprising that the rate constant of the reaction of two charged species practically does not depend on an ionic strength. For example, the work [29] gives $k_7 = 6.3 \times 10^5 \text{ M}^{-1} \text{ s}^{-1}$ at $\mu = 0$ whereas the authors of [23] have obtained $k_7 = 6.6 \times 10^5 \text{ M}^{-1} \text{ s}^{-1}$ in 1–7 M H_2SO_4 . However, in the recent work [33] the estimation of upper limit to the rate constant $k_7 \leq 10^4 \text{ M}^{-1} \text{ s}^{-1}$ is obtained which is in good agreement with our value. The k_8 values change from 3×10^3 [34] to $3.6 \times 10^2 \text{ s}^{-1}$ [19,33]. Our larger k_8 value can be determined by the high ionic strength and the uncontrollable impurities in solution. The strong dependence of the k_8 on the ionic strength is discussed in [33].

3.2. Photolysis of $\text{Na}_2\text{S}_2\text{O}_8$ solutions in the presence of DMF

The flash photolysis of the aqueous solutions of $\text{Na}_2\text{S}_2\text{O}_8$ in the presence of DMF gives rise to supplement intermediate absorption whose band maximum is shifted towards the blue region in comparison with the $\text{SO}_4^{\bullet-}$ band. In Fig. 1 the spectrum of this absorption (curve 2) is obtained upon photolysis of solutions with high DMF concentration (10^{-2} M). In this case, the primary radical $\text{SO}_4^{\bullet-}$ is rapidly and completely converted into a new species. The new spectrum corresponds to the published [10,11] one of the $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical which results from the reaction

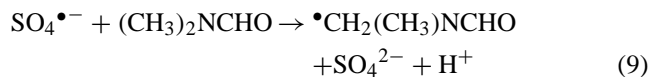


Fig. 5 shows the kinetic curves at various wavelengths for substantially lower DMF concentration ($4 \times 10^{-4} \text{ M}$) at which we observed the formation and decay of a new intermediate absorption. In this case, the signal to measure is a superposition of absorption of $\text{SO}_4^{\bullet-}$ and $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radicals. When the wavelength is more than 500 nm the $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical does not actually absorb (Fig. 1). Therefore, to obtain the kinetic curves describing the behavior of this species at a given wavelength, it is necessary to subtract the normalized curve at 500 nm (curve 2 in Fig. 5) from the experimental curve at this wavelength (curve 1 in Fig. 5). Normalization is determined by the $\text{SO}_4^{\bullet-}$ spectrum obtained in the absence of DMF.

The kinetic curves of the $\text{SO}_4^{\bullet-}$ radical anion decay in the presence of DMF were determined by the pseudo-first and second order processes related to reactions (7–9) and (5). To demonstrate the process of $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical formation (curve 3 in Fig. 5), the lower laser flash intensity was used. In this case, the $\text{SO}_4^{\bullet-}$ radical anion

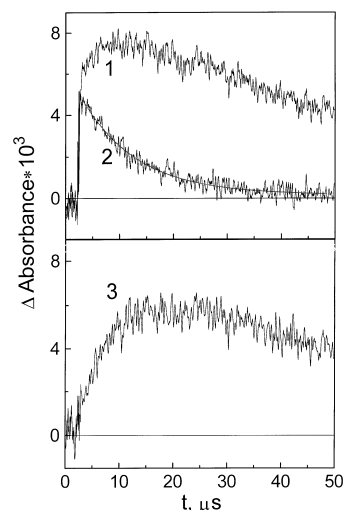


Fig. 5. Kinetic curves of a change in intermediate absorption upon photolysis of sodium persulfate (0.3 M) in oxygen-free aqueous solution with added DMF ($4 \times 10^{-4} \text{ M}$). Curve 1 — the experimental curve at 380 nm ($\text{SO}_4^{\bullet-} + \bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$) curve 2 and solid line — the experimental curve at 500 nm ($\text{SO}_4^{\bullet-}$ only) and first order fit; curve 3 — the difference in curves (1) and (2) (having regard to the equality of $\text{SO}_4^{\bullet-}$ extinction coefficients at 380 and 500 nm) — the kinetic curve for only ($\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$).

decay presumably defines by the pseudo-first order reactions. This is confirmed by the first order fit of $\text{SO}_4^{\bullet-}$ decay curve (dashed line in Fig. 5).

To determine k_9 from the initial part of kinetic curves at a wavelength of 500 nm we measured k_{obs} with respect to the initial optical density (Fig. 6a). The cut-off on the ordinate depends linearly on DMF concentration (Fig. 6b)

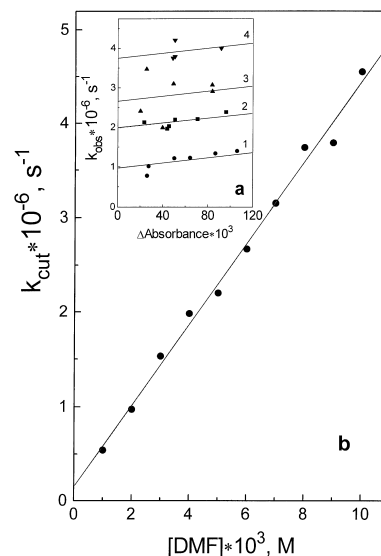


Fig. 6. Reaction between $\text{SO}_4^{\bullet-}$ radical anion and DMF. (a) The dependence of k_{obs} of $\text{SO}_4^{\bullet-}$ radical anion decay on the initial optical density at 500 nm for various DMF concentrations. 1–4: DMF concentration (0.002, 0.004, 0.006, 0.008 M, respectively). (b) The dependence of the cut-off on the ordinate in (a) on DMF concentration.

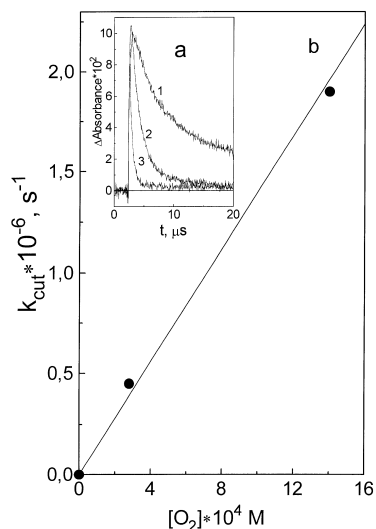


Fig. 7. Kinetic curves (a) and k_{cut} (b) of $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ decay at 380 nm for various concentrations of oxygen in solution. Photolysis (308 nm) of $\text{Na}_2\text{S}_2\text{O}_8$ (0.3 M) in presence of 0.01 M DMF. 1–3, oxygen concentration being 0, 2.5×10^{-4} M and 1.4×10^{-3} M, respectively.

and the rate constant k_9 , determined from the slope is $(4.3 \pm 0.2) \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$.

At fairly high DMF concentrations ($\geq 10^{-2}$ M) reaction (9) becomes almost the only channel of $\text{SO}_4^{\bullet-}$ radical anion decay. This allows one to determine the extinction coefficient of $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical by comparing the initial absorption at 500 and 380 nm. The extinction coefficient is $\epsilon^{380} = (1700 \pm 200) \text{ M}^{-1} \text{ cm}^{-1}$ which is in fair agreement with the value $1600 \text{ M}^{-1} \text{ cm}^{-1}$ given in [10] for water at pH=5 and with the value of $2500 \text{ M}^{-1} \text{ cm}^{-1}$ obtained for the frozen DMF matrix [11].

3.3. $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radicals decay

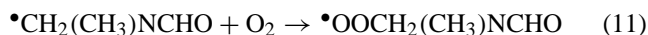
The rate of $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical decay (k_{obs}) in the oxygen-free solutions depends linearly on its initial concentration. Therefore, the decay of radicals is made by the second-order reaction, i.e. either recombination or disproportion



The $2k_{10}/\epsilon^{380}$ value was found to be $(1.55 \pm 0.09) \times 10^6 \text{ cm s}^{-1}$. Using the extinction coefficient for the absorption band of the $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical ($\epsilon^{380} = 1700 \text{ M}^{-1} \text{ s}^{-1}$), we obtain the value of $2k_{10} = (2.64 \pm 0.16) \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$ for ionic strength $\mu = 0.9$ M. The results of work [12] for the solutions with $\mu = 0.08$ and pH=1.5 using the same value of ϵ^{380} give $2k_{10} = (4.6 \pm 1.1) \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$ which is somewhat larger than that in a given paper. The difference can be determined by the increase of the solution viscosity at the high $\text{Na}_2\text{S}_2\text{O}_8$ concentration (0.3 M) and the weak

influence of ionic strength on the reaction of neutral radicals which have the dipole moments.

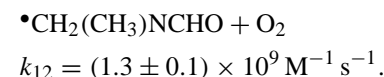
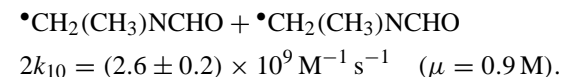
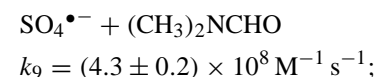
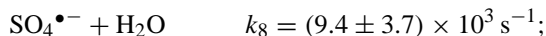
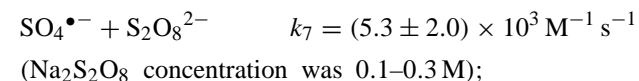
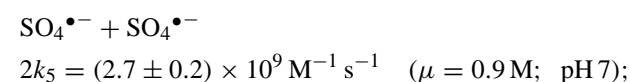
The rate of $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radical decay depends substantially on the concentration of oxygen contained in water (Fig. 7a). To measure the rate constant of the reaction between these radicals and oxygen, we determined the dependence of k_{obs} on initial radical concentration and then the dependence of obtained cut-off (k_{cut}) on the ordinate on the concentration of dissolved oxygen (Fig. 7b). Probably, the $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ radicals, as many organic radicals [35], transform in the presence of oxygen into less active peroxide radicals



The slope angle in Fig. 7b may be used to determine that $k_{12} = (1.3 \pm 0.1) \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$. Note that the value of this constant falls within the range of the characteristic values of the rate constant of the formation of peroxide radicals $(0.5\text{--}5) \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$ [35].

4. Conclusions

$\text{SO}_4^{\bullet-}$ radical anion is formed by the photolysis of $\text{S}_2\text{O}_8^{2-}$ at 308 nm with the quantum yield of $\phi^{308} = 0.55 \pm 0.05$. $\text{SO}_4^{\bullet-}$ reacts with DMF to form the radical $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ having an absorption band with a maximum at 380 nm ($\epsilon^{380} = (1700 \pm 200) \text{ M}^{-1} \text{ cm}^{-1}$). Several rate constants of $\text{SO}_4^{\bullet-}$ and $\bullet\text{CH}_2(\text{CH}_3)\text{NCHO}$ have been measured. These are:



The DMF may be used as an effective trap of active radicals (e.g., $\bullet\text{OH}$ radical) which cannot be recorded directly due to the absence of optical absorption in the available spectrum range.

Acknowledgements

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